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4,6-Dibromo-2,3-dimethylphenol

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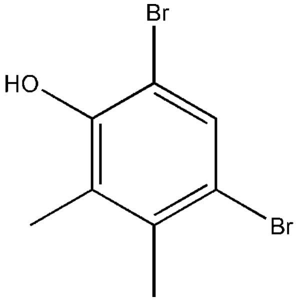
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.037; wR factor = 0.090; data-to-parameter ratio = 22.1.

The molecule of the title compound, $C_8H_8Br_2O$, is approximately planar with a maximum deviation of 0.063 (1) Å for one of the Br atoms. In the crystal, adjacent molecules are joined intermolecular O—H···O hydrogen bonds, forming chains parallel to [010]. The structure also features a short Br···Br interaction of 3.362 (1) Å.

Related literature

For the synthesis, see: Lai *et al.* (1993). For a related structure, see: Bringmann & Messer (2001).



Experimental

Crystal data

$C_8H_8Br_2O$
M_r = 279.96
Monoclinic, $P2_1$
a = 7.3604 (5) Å
b = 4.4310 (6) Å
c = 14.0245 (10) Å
β = 92.482 (1)°
V = 456.96 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 8.81 mm^{-1}
T = 298 K
0.16 × 0.12 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.333, T_{max} = 0.473
5557 measured reflections
2250 independent reflections
1882 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.090
S = 0.99
2250 reflections
102 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack (1983), 1275 Friedel pairs
Flack parameter: 0.02 (2)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O1^{i}	0.82	2.25	2.913 (4)	139

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+2$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5106).

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supporting information

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4,6-Dibromo-2,3-dimethylphenol

Qiaoru Liu, Jungang Wang, Weijian Xue and Qi Li

S1. Comment

In the title compound,*C*~8~H~8~Br~2Õ, the adjacent molecules are molecules are joined togethe by the O1—H1···O1 (-*x*, *y* - 1/2, 2 - *z*) hydrogen bond, forming a one-dimensional chain running parallel to the [010] direction(Table 1 and Figure 2). Also Br···Br interaction was observed in (I) with a distance of 3.362 (1) Å between them All the bond lengths and angles are similar to the reported compound (Bringmann *et al.*, 2001).

S2. Experimental

The title compound, synthesized by 2,3-dimethyl phenylamine through three steps such as bromination, diazotization-bromination-hydrolysis reaction.The operating process was based on the literarure (Lai *et al.*, 1993) and made some improvement.

Firstly, 1-amino-4-bromo-2,3-dimethylbenzene was prepared from 2,3-dimethyl phenylamine as described in the literarure(Lai *et al.*, 1993). Then treatment as follows: Sodiumnitrite (1.75 g, 25 mmol) in water (10 ml) was added dropwise into the rapidly stirring mixture of 40% hydrogen bromide (15 ml) containing l-amino-2,3-dimethylbenzene (5.00 g, 25 mmol). The mixture was kept in an ice-bath stiring for 2 h, while the temperature was kept below 5°C by the addition of pieces of ice. Then added 1.97 g (14 mmol) cuprous bromide which was pretreatmented by refluxing with 10 ml 40% hydrogen bromide solution for 1 h. After the addition the mixture was heated refluxing for an additional 1 h, and then cooled to room temperature, extract by methylenechloride. The organic layer was washed by water, dried by anhydrous natriumsulfate, evaporated under reduced pressure and chromatographed on silica gel with hexane as the eluent. The title compound was obtained as needle crystal solid 1.82 grams. Yield was 26%. Colorless needle-like single crystals suitable for X-ray diffraction studies were obtained by slow evaporation of a solution of the title compound in chloroform: methanol (3: 1) at room temperature.

S3. Refinement

In (I), all carbon H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 Å and *U*\ĩso\~(H) = 1.2*U*\~eq\~(C) for aromatic H atoms, and C—H = 0.96 Å and *U*\ĩso\~(H) =1.5*U*\~eq\~(C) for methyl H atoms. H1 atom was found first from the difference map and placed at its ideal position with the O—H=0.82Å and U\ĩso\~(H)=1.5U\~eq\~(O). The Friedel pairs is 1275.

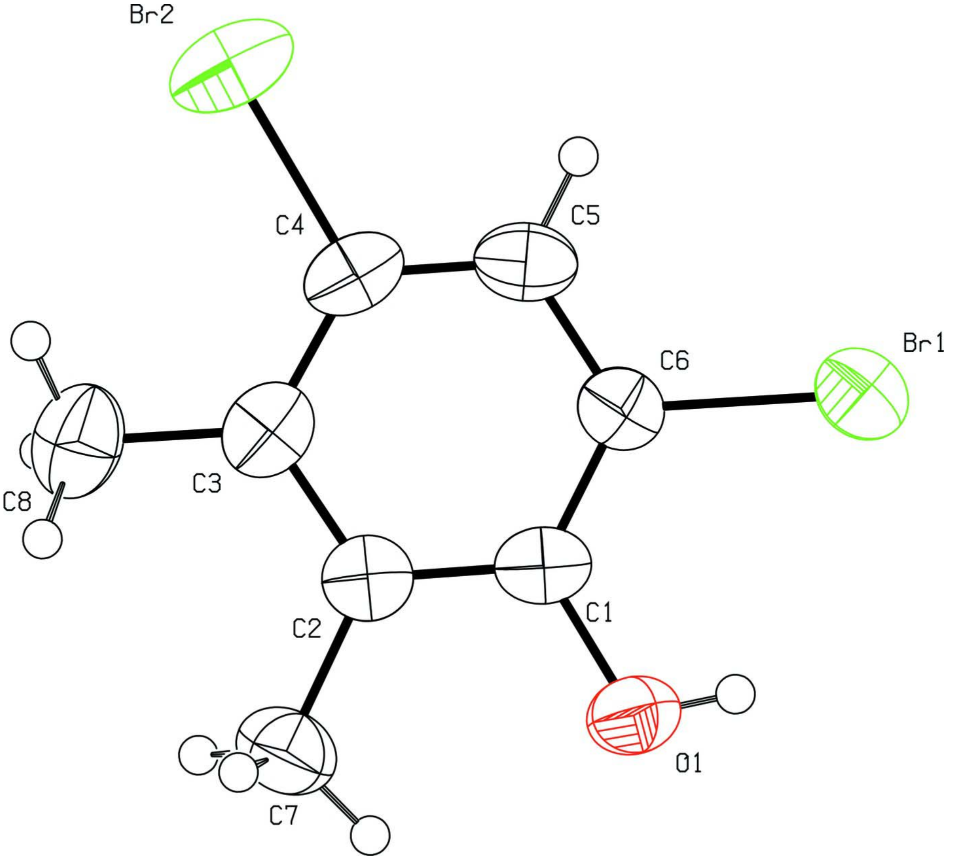

Figure 1

The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

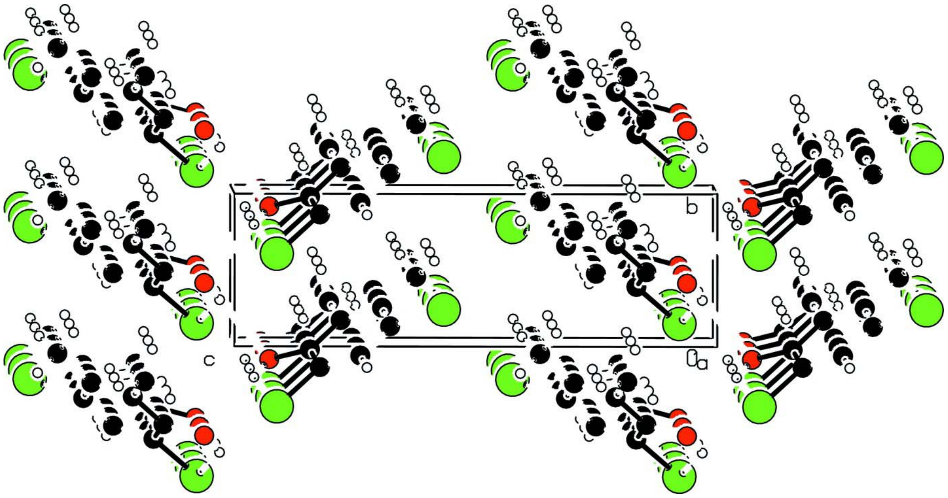

Figure 2

Part of the crystal packing, showing the formation of the one-dimensional chain in (I) by the O1—H1···O1(-*x*, *y* - 1/2, 2 - *z*) hydrogen bond.

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Crystal data

$C_8H_8Br_2O$
$M_r = 279.96$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 7.3604$ (5) Å
$b = 4.4310$ (6) Å
$c = 14.0245$ (10) Å
$\beta = 92.482$ (1)°
$V = 456.96$ (8) Å^3
$Z = 2$
$F(000) = 268$
$D_x = 2.035$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2355 reflections
$\theta = 2.8$–24.5°
$\mu = 8.81$ mm^{-1}
$T = 298$ K
Needle, colorless
0.16 × 0.12 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.333$, $T_{\max} = 0.473$
5557 measured reflections
2250 independent reflections
1882 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.042$
$\theta_{\max} = 28.3°$, $\theta_{\min} = 2.8°$
$h = -9 \rightarrow 9$
$k = -5 \rightarrow 5$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.090$
$S = 0.99$
2250 reflections
102 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.57$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}
Absolute structure: Flack (1983), 1275 Friedel pairs
Absolute structure parameter: 0.02 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.87217 (5)	−0.34219 (12)	0.92038 (3)	0.05521 (15)
Br2	0.86105 (8)	0.29262 (15)	0.57447 (4)	0.0790 (2)
C1	0.5729 (5)	0.0139 (10)	0.8491 (3)	0.0436 (9)

C2	0.4789 (5)	0.2000 (10)	0.7846 (3)	0.0458 (10)
C3	0.5600 (6)	0.2866 (12)	0.6995 (3)	0.0496 (9)
C4	0.7350 (6)	0.1753 (13)	0.6843 (3)	0.0516 (9)
C5	0.8255 (5)	−0.0114 (11)	0.7471 (3)	0.0498 (10)
H5	0.9405	−0.0835	0.7342	0.060*
C6	0.7440 (5)	−0.0922 (9)	0.8300 (3)	0.0429 (9)
C7	0.2920 (6)	0.3103 (13)	0.8071 (4)	0.0621 (12)
H7A	0.2479	0.1971	0.8597	0.093*
H7B	0.2110	0.2837	0.7522	0.093*
H7C	0.2978	0.5204	0.8237	0.093*
C8	0.4589 (8)	0.4836 (14)	0.6281 (4)	0.0684 (14)
H8A	0.5442	0.5917	0.5911	0.103*
H8B	0.3849	0.6245	0.6610	0.103*
H8C	0.3828	0.3605	0.5866	0.103*
O1	0.4886 (4)	−0.0551 (8)	0.9326 (2)	0.0533 (8)
H1	0.5505	−0.1777	0.9634	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0508 (2)	0.0528 (2)	0.0620 (3)	0.0041 (2)	0.00077 (17)	−0.0045 (2)
Br2	0.0927 (4)	0.0918 (4)	0.0547 (3)	−0.0172 (3)	0.0285 (3)	−0.0037 (3)
C1	0.043 (2)	0.0419 (19)	0.046 (2)	−0.0093 (17)	0.0072 (16)	−0.0128 (19)
C2	0.047 (2)	0.042 (3)	0.049 (2)	−0.0074 (17)	0.0016 (16)	−0.0067 (17)
C3	0.061 (2)	0.044 (2)	0.043 (2)	−0.012 (2)	−0.0015 (18)	−0.007 (2)
C4	0.059 (2)	0.054 (2)	0.043 (2)	−0.015 (2)	0.0118 (16)	−0.004 (2)
C5	0.045 (2)	0.049 (2)	0.056 (3)	−0.006 (2)	0.0103 (18)	−0.014 (2)
C6	0.043 (2)	0.042 (2)	0.043 (2)	−0.0011 (17)	−0.0010 (16)	−0.0099 (18)
C7	0.046 (2)	0.066 (3)	0.074 (3)	0.004 (2)	0.007 (2)	0.001 (3)
C8	0.089 (4)	0.063 (3)	0.052 (3)	−0.001 (3)	−0.006 (3)	0.002 (3)
O1	0.0545 (17)	0.0582 (19)	0.0482 (18)	0.0023 (15)	0.0143 (13)	0.0007 (15)

Geometric parameters (Å, º)

Br1—C6	1.903 (4)	C5—C6	1.379 (6)
Br2—C4	1.905 (4)	C5—H5	0.9300
C1—C6	1.381 (5)	C7—H7A	0.9600
C1—O1	1.383 (5)	C7—H7B	0.9600
C1—C2	1.387 (6)	C7—H7C	0.9600
C2—C3	1.411 (6)	C8—H8A	0.9600
C2—C7	1.507 (6)	C8—H8B	0.9600
C3—C4	1.404 (7)	C8—H8C	0.9600
C3—C8	1.501 (7)	O1—H1	0.8200
C4—C5	1.361 (7)		

C6—C1—O1	122.4 (4)	C5—C6—Br1	119.5 (3)
C6—C1—C2	120.6 (4)	C1—C6—Br1	119.9 (3)
O1—C1—C2	117.1 (3)	C2—C7—H7A	109.5

C1—C2—C3	119.8 (4)	C2—C7—H7B	109.5
C1—C2—C7	119.4 (4)	H7A—C7—H7B	109.5
C3—C2—C7	120.9 (4)	C2—C7—H7C	109.5
C4—C3—C2	117.2 (4)	H7A—C7—H7C	109.5
C4—C3—C8	122.4 (4)	H7B—C7—H7C	109.5
C2—C3—C8	120.4 (4)	C3—C8—H8A	109.5
C5—C4—C3	122.8 (4)	C3—C8—H8B	109.5
C5—C4—Br2	116.6 (3)	H8A—C8—H8B	109.5
C3—C4—Br2	120.6 (4)	C3—C8—H8C	109.5
C4—C5—C6	119.0 (4)	H8A—C8—H8C	109.5
C4—C5—H5	120.5	H8B—C8—H8C	109.5
C6—C5—H5	120.5	C1—O1—H1	109.5
C5—C6—C1	120.6 (4)		

C6—C1—C2—C3	−1.2 (6)	C2—C3—C4—Br2	−177.2 (3)
O1—C1—C2—C3	178.0 (4)	C8—C3—C4—Br2	4.2 (7)
C6—C1—C2—C7	179.6 (4)	C3—C4—C5—C6	−1.1 (7)
O1—C1—C2—C7	−1.3 (6)	Br2—C4—C5—C6	177.0 (3)
C1—C2—C3—C4	0.3 (6)	C4—C5—C6—C1	0.2 (6)
C7—C2—C3—C4	179.6 (4)	C4—C5—C6—Br1	−177.9 (3)
C1—C2—C3—C8	178.9 (4)	O1—C1—C6—C5	−178.2 (4)
C7—C2—C3—C8	−1.8 (7)	C2—C1—C6—C5	0.9 (6)
C2—C3—C4—C5	0.8 (7)	O1—C1—C6—Br1	−0.1 (5)
C8—C3—C4—C5	−177.8 (5)	C2—C1—C6—Br1	179.0 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O1^{i}	0.82	2.25	2.913 (4)	139
O1—H1···Br1	0.82	2.57	3.108 (3)	124
C8—H8*A*···Br2	0.96	2.70	3.200 (6)	113

Symmetry code: (i) −*x*+1, *y*−1/2, −*z*+2.